

Matagorda Bay Mitigation Trust 2022-2023 Funding Cycle  
RFP # 2022-2023-1

**Title: Reproductive & Developmental Toxicity of “Forever Chemicals” to  
Matagorda Bay’s prey fishes**

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Q2 January 2024 Progress Report

**Q2 Update:**

**We have completed the sampling needed to characterize the PFAS profile of Matagorda Bay as part of Phase 1. As of now, we have completed all environmental sampling at the four proposed sampling locations (see Figure 1). Water and sediment samples have all been prepared for PFAS analysis. All sediment samples have been sent out to analytical chemistry labs for PFAS quantification. All water samples are currently being analyzed at UT MSI Core facility for PFAS quantification.**

**Sample Collection:**

During Q2, we collected samples from the last of our four sampling locations (Figure 1), downstream of the Chocolate Bay Wastewater Treatment Plant (Figure 2). At were done with the 3 previously sampled locations, samples were taken in triplicate from 3 sites along a 20 meter transect (Figure 2). At each sampling site along the transects, a total of three water samples were collected in PFAS-free bottles. Following water collection, five 5cm sediment cores were taken at the same location and placed in a PFAS-free bottle. In addition to sample collection, each site had the following recorded: latitude + longitude, salinity, dissolved oxygen, pH, and temperature.

In total, 27 water samples and 9 composite sediment samples were collected downstream of the Chocolate Bay Wastewater Treatment Plant (Figure 2) Following collection, all samples were immediately placed on ice and transported to the University of Texas at Austin Marine Science Institute (UT MSI). Samples were placed at -20°C until processing for analysis. Storage at -20°C is the most accepted pre-analysis storage, and has been shown to keep PFAS stable for more >6 months prior to analysis<sup>1</sup>.

**Sample Analyses:**

All sediment samples (9 per site, 4 sites total) have been sent to either SGS AXYS Analytical Services, LTD or Michigan State University PFAS Centre’s Analytical Chemistry Lab for PFAS Analysis. These samples will be extracted and analyzed following EPA Draft Method 1633 “821-D-21-001: Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS” (Figure 3), a method that identifies 40 PFAS (Table 1). SGS AXYS Analytical Services, LTD have received accreditation for EPA Method

1633 from the US Department of Defense. Michigan State University PFAS Centre's Analytical Chemistry Lab is an established PFAS testing and research centre. This analysis will provide a full profile of the 40 most prevalent PFAS (Table 1) and will provide the necessary data to create a PFAS profile for Matagorda Bay. These data will also provide PFAS profiles specific to each point source identified. Samples are also being assessed for dissolved organic carbon (DOC), as DOC is known to significantly influence bioavailability of PFAS.

Water samples (9 per site; i.e., one pre transect) have been prepared for analysis following EPA Method 531.1 "*Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid*" (Figure 3) at UT MSI. This method will detect the presence of 22 PFAS (Table 2). Water samples have all undergone PFAS extraction using Solid Phase Extraction (SPE) following Method 531.1 (Figure 3). Briefly, each water sample was defrosted from -20°C, spiked with the EPA 537.1 Method isotopically-labelled internal standards, run through the PFAS-free resin SPE columns (Bond Elut-LMS, 500mg 6mL; Agilent Technologies) recommended by the EPA, and placed at -20°C until elution. Samples were then eluted with methanol, spiked with the EPA 537.1 Method isotopically-labelled surrogate standard, reconstituted to 1 mL (96% Methanol: 4% DI Water), and sent to the Analytical Core Laboratory at UT MSI for PFAS analysis on the IM Q-TOF LC-MS. The Analytical Core Laboratory at UT MSI has developed the LC and QTOF methods following EPA Method 531.1, confirmed PFAS are retained, and confirmed they can be correctly identified. The Analytical Core Laboratory has also determined detection limits and have performed the necessary standard curve calibrations. All samples are currently being analyzed. These analyses will provide the necessary data to characterize the PFAS present in water samples taken from each sampling location in Matagorda Bay.

### **Preparation for Phases 2 and 3:**

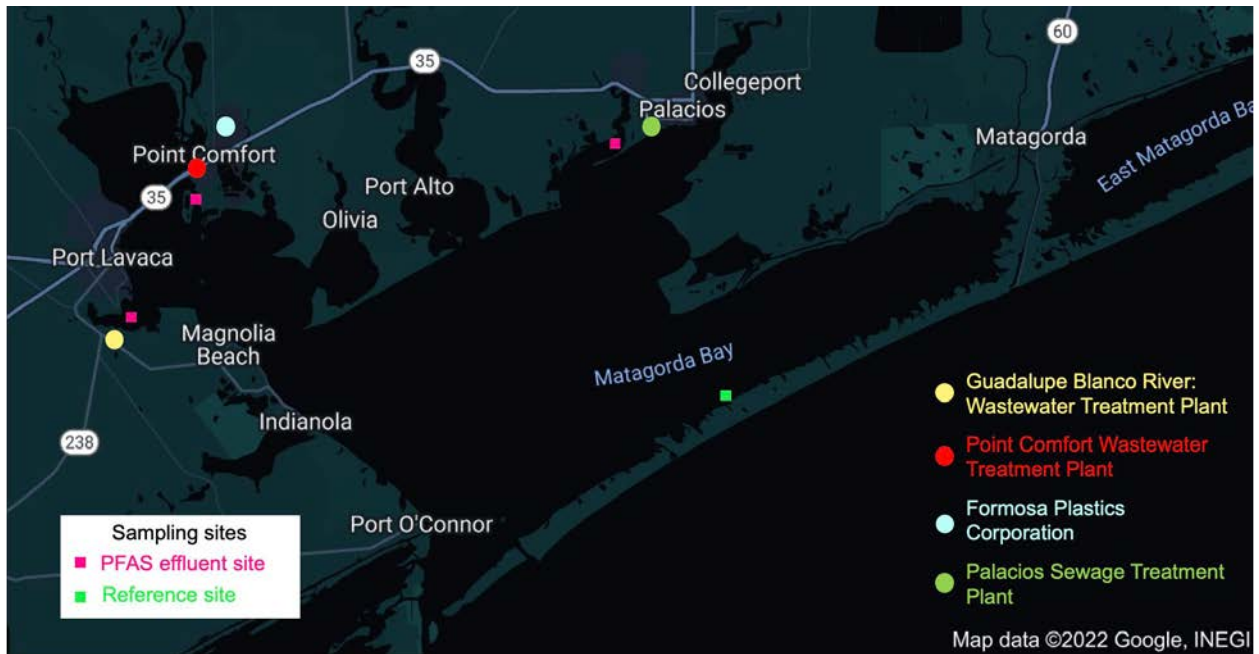
We have also now identified locations for seining for collection of sheepshead minnows at each site (Q1 Report; Figure 2). Sheepshead minnows will be collected for analysis of PFAS body burdens using EPA Method 1633 from these locations for Phase 2. These same sites will be used to collect sheepshead minnows for transport to UT MSI for the chronic PFAS exposures and reproductive / fecundity studies detailed in Phase 3.

### **References:**

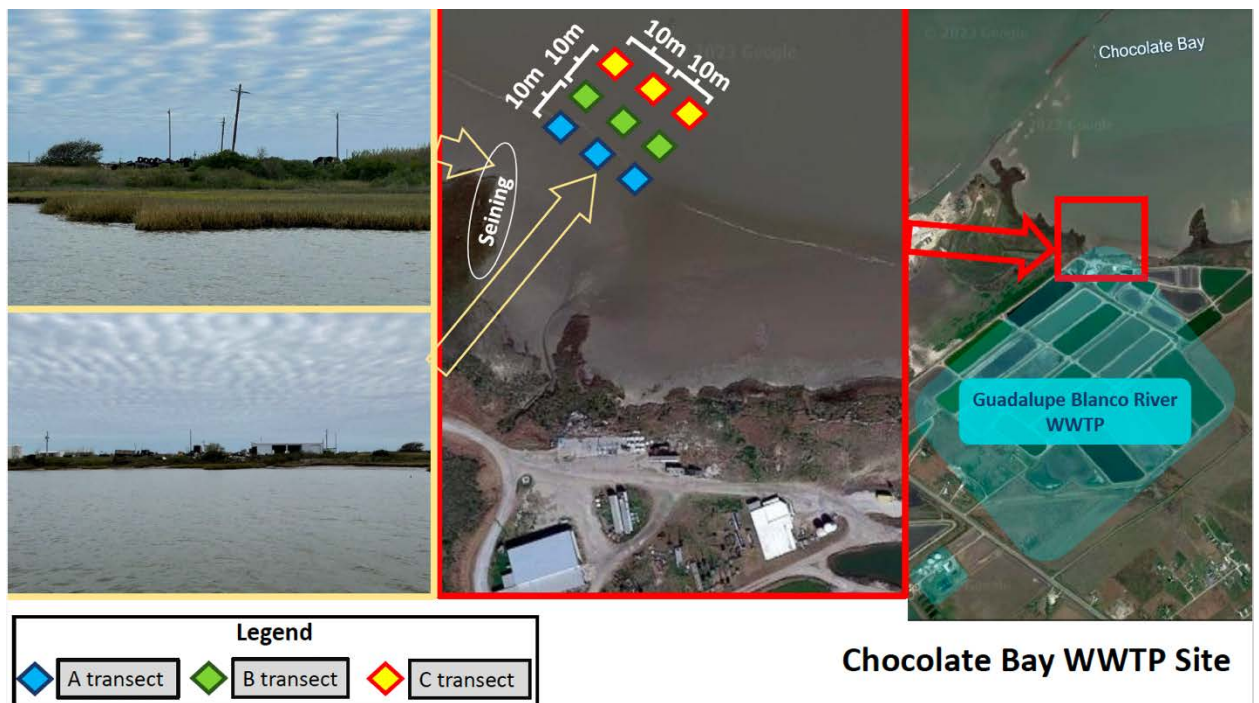
<sup>1</sup>Woudneh, Million B., Bharat Chandramouli, Coreen Hamilton, and Richard Grace. "Effect of sample storage on the quantitative determination of 29 PFAS: observation of analyte interconversions during storage." *Environmental Science & Technology* 53, no. 21 (2019): 12576-12585.

<sup>2</sup><https://www.sgsaxys.com/2021/09/14/epa-announces-availability-of-epa-1633-draft-pfas-method-developed-by-sgs-axys-sgs-axys-continues-to-expand-range-of-pfas-testing-methods/>

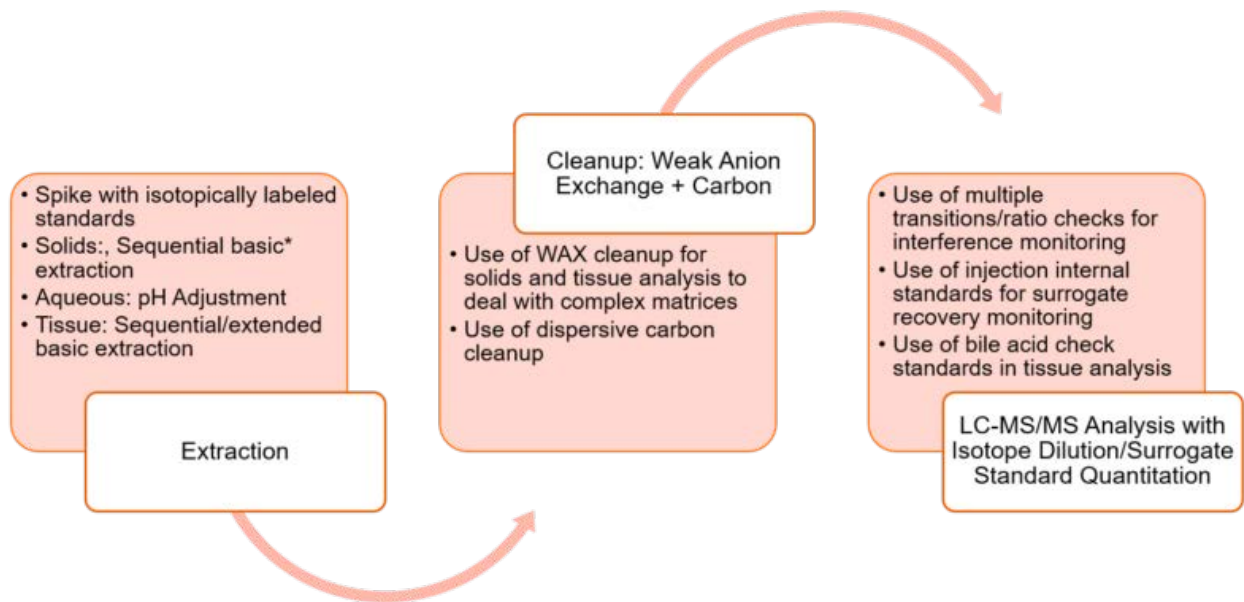
**Figures:**



**Figure 1.** Expected point sources for introduction of PFAS into the Matagorda Bay system and the proposed sampling sites for characterization of PFAS in the Bay.



**Figure 2.** Site sampled as an expected point source of PFAS for PFAS characterization of Matagorda Bay near chocolate Bay Wastewater Treatment Plant.



**Figure 3.** Extraction and analysis methods used for water and sediment samples for PFAS analysis using EPA Methods 1633 and 537.1. Schematic provided by SGS AXYS Analytical Services, LTD<sup>2</sup>.

**Tables:**

FAMILY	ANALYTE	TYPICAL REPORTING LIMITS
PFCA	PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnA, PFDoA, PFTrDA, PFTetraDA	<ul style="list-style-type: none"> <li>• 0.4-1.6 ng/L water</li> <li>• 0.04-0.16 ng/g solid</li> <li>• 0.1-0.4 ng/g tissue</li> <li>• 0.1-0.4 ng/mL serum</li> <li>• 10-40 ppb AFFF</li> </ul>
PFSA	PFBS, PFPeS, PFHxS, PFHpS, PFOS, PFNS, PFDS, PFDoS	<ul style="list-style-type: none"> <li>• 0.4 ng/L water</li> <li>• 0.04 ng/g solid</li> <li>• 0.1 ng/g tissue</li> <li>• 0.1 ng/mL serum</li> <li>• 10 ppb AFFF</li> </ul>
FTS and FTCA	4:2, 6:2 and 8:2 FTS, 3:3, 5:3 and 7:3 FTCA	<ul style="list-style-type: none"> <li>• 3.2- 10 ng/L water</li> <li>• 0.32 – 1 ng/g solid</li> <li>• 0.8 – 2.5 ng/g tissue</li> <li>• 40-250 ppb AFFF</li> </ul>
Sulfonamides	EtFOSAA, MeFOSAA, PFOSA, EtFOSA, MeFOSA, EtFOSE and MeFOSE	<ul style="list-style-type: none"> <li>• 0.4-4 ng/L water</li> <li>• 0.04-0.4 ng/g solid</li> <li>• 0.1-1 ng/g tissue</li> <li>• 10-100 ppb AFFF</li> </ul>
Ether carboxylates and sulfonates	HFPO-DA (GEN-X), ADONA, F-53B, NFDHA, PFMBA, PFMPA, PFEESA	<ul style="list-style-type: none"> <li>• 0.4-1.6 ng/L water</li> <li>• 0.04 – 0.16 ng/g solid</li> <li>• 0.1-0.4 ng/g tissue</li> <li>• 10-40 ppb AFFF</li> </ul>

Table 1. PFAS to be analyzed using EPA Method 1633 for sediment (i.e., solid) samples taken from each sampling site in Matagorda Bay. Table provided by SGS AXYS Analytical Services, LTD<sup>2</sup>.

Analyte	Internal Standard Reference
PFBS	2
PFHxA	1
HFPO-DA	1
PFHpA	1
PFHxS	2
ADONA	1
PFOA	1
PFOS	2
PFNA	1
9Cl-PF3ONS	2
PFDA	1
NMeFOSAA	3
PFUnA	1
NEtFOSAA	3
11Cl-PF3OUdS	2
PFDoA	1
PFTTrDA	1
PFTA	1
<sup>13</sup> C2-PFHxA	1
<sup>13</sup> C3-HFPO-DA	1
<sup>13</sup> C2-PFDA	1
d5-NEtFOSAA	3
<sup>13</sup> C2-PFOA– IS#1	-
<sup>13</sup> C4-PFOS– IS#2	-
d3-NMeFOSAA–IS#3	-

Table 2. PFAS to be analyzed using EPA Method 537.1 for water samples taken from each sampling site in Matagorda Bay.